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Optical method for characterization of particulate systems and device for carrying out the method

The invention concerns an optical method for characterization of particulate systems, in particular for clean room monitoring, with which the particles present in a particulate system, for example a clean room, can be detected in respect of quantity and size and at the same time information about the identity of the particles can be provided. The invention also concerns a device for carrying out the method. The method and the associated device make it possible for example to implement preventative quality assurance in clean rooms.

Because of the increasing miniaturization of products, the electronics industry involves very high demands in terms of the purity and cleanliness of the gases involved in the production process. If the product structures are of the same order of magnitude as the diameters of the particles to be encountered in the gases, they act as "killer particles" in the production process. The cleanliness demands in terms of the room air in the production processes in the electronics industry are therefore becoming more and more strict and require knowledge about the origin,

movement and substance composition of the particles. At the present time in principle two different methods are used for particle measurement and particle analysis separately from each other.

So-called particle counters are used for determining the level of particle concentration in the clean room air. These involve measuring devices which are capable of continuously measuring an air sample from the clean room. These can be measuring devices which by way of an optical method can detect particles to a size of 0.1 μm and can attribute them to given size categories. Special particle counters, so-called condensation nucleus counters, permit the measurement of particles to a size of 0.05 μm. That is made possible by virtue of the fact that particles are increased in size due to condensation of a liquid and are measured thereafter.

The particle counters serve exclusively for counting the particles, and analysis of the material composition of the particles is not possible. The measured particles are also no longer available for later analysis with other measurement systems as the sample volume is abandoned after flowing through the measuring device. It is therefore necessary to take another sample again, for further analysis procedures.

There are numerous measuring devices for particle analysis, which make it possible to establish the material composition of particles. Those measuring devices operate inter alia on the basis of the principle of electron-laser spectroscopy. The measurement systems are generally disposed in separate laboratories because generally they are not suitable for a clean room situation and they require very difficult sample preparation. Direct analysis of the particles in the clean room air is not possible with those devices.

At the present time there is a method in the course of development, which can analyze both the number and size of particles and also the particle composition. That method is based on the mass-spectroscopic analysis of particles which were ionized by means of UV-lasers. That technology however is not suitable for a clean room situation by virtue of

the oil pumps used. In addition the size of the measuring unit does not permit mobile use thereof and it will probably be very expensive.

The object of the present invention is to develop a method and an associated device for detecting in respect of quantity and size and simultaneously determining the identity of the particles present in a particulate system, in particular in a clean room, which permits automatic online operation, which is suitable for a clean room situation, which is inexpensive and industry-standardized and which can be put to mobile use.

In accordance with the invention that object is attained by an optical method for characterization of particulate systems as set forth in claim 1 and a device for carrying out the method as set forth in claim 7.

In accordance with the invention an air flow comprising the ambient air is guided at a defined speed through a particle feeder past a first scattered light measuring unit and the scattered light is detected, the speed of the particle is then reduced and the particle which is moved at the reduced speed is identified in the air flow in an identification unit by means of interaction with monochromatic light.

It is possible for the first time with that method to quantitatively detect the particles present in a clean room and at the same time to provide information about the identity of the particles. This therefore provides the clean room operators with a tool which for the first time makes it possible to implement preventative quality assurance and thus to very substantially satisfy the rising demands in terms of cleanliness of the room air used in the electronic production process.

In a preferred configuration of the invention the optical system of the identification unit, the spectrometer unit, is triggered by way of an electronic control by the scattered light measuring unit. By virtue thereof, it is possible in the analysis step following the particle size determining operation, if required, to identify only particles in a preselected range, that is to say for example only particles of a given diameter or in a given diameter range.

The selection criteria can be determined and selected under software control by means of the electronic control. Such a selection option is particularly advantageous in terms of using the identification unit in particle-rich environments.

In accordance with the invention, identification of the particles is effected by combined laser-Raman spectroscopy which, with a short exposure time, by virtue of the use of high-power light sources, strong-light optics and in particular by virtue of foregoing high levels of optical resolution (normally 4 cm^{-1}, here 12-24 cm^{-1}), affords results which are usable for automated evaluation. The low level of spectral resolution is used to advantage.

The reduction of the speed of the particles to a residence time of about 1 ms to about 1 s in the second laser beam serves to obtain vibration spectra which show all spectral features and which are suitable for automated evaluation. Signals which are obtained without the speed reduction of the particle are not sufficient for identification in the predominant number of cases as the noise increases greatly and therefore automated evaluation of the spectra becomes impossible.

The Raman spectra obtained are electronically filtered and investigated for spectral features (peaks) and the peak table obtained is finally compared to a database which contains corresponding reference tables and the substance identified.

The device according to the invention comprises module units which include at least the following elements:

- an optical unit for determining the size and number of particles in an air flow from the ambient air,
- a particle brake,
- an optical identification unit for the moved particles contained in the air flow, comprising corona discharge, excitation laser and spectrometer unit, and
- an electronic control.

The modular structure of the system is an essential point of view as on the one hand it permits further development and application in wider areas of use while on the other hand it permits the replacement of individual modules by other suitable ones according to the properties of the particles to be identified. Thus for example different demands can be made on the spectrometer unit, depending on whether organic impurities or biotic particles have to be identified. For example, a resonance Raman module would be used for the identification of biotic particles, either jointly with the Raman module or instead of the Raman module.

In addition the system is preferably in the form of a mobile unit, involving dimensions of a maximum of about 1 x 2 x 1 m and of a weight of about 40 kg so that use can take place directly at the location to be sampled and the samples do not have to be sent to analysis laboratories. That for example makes it possible to implement preventative clean room monitoring.

The light source of the identification unit is preferably a narrow-band light source, preferably a monochromatic light source. The spectrometer unit of the identification unit is preferably formed from an NIR multichannel spectrometer. The multichannel spectrometer preferably has about 255 detectors and preferably has a measuring range of approximately 900 – 1,900 nm. That technology is inexpensive and permits the desired small dimensions for the entire measuring device.

By virtue of the only short available measuring times of between 1 ms and 1 s, particular demands are to be made on the light source. A particularly suitable light source has been found to be a narrow-band light source, preferably a monochromatic light source with a high power output. It is however also possible to use other suitable laser light sources, for example multimode laser diodes, wide-band laser diodes and pulsed laser light sources.

By virtue of the renunciation of resolution and the use of simple components, this new technology combines laser spectroscopy with the simplicity and convenience of other optical methods, for example NIR

spectroscopy. In contrast to FT spectroscopy that permits the very short measurement times specified.

The electronic control, on the basis of predetermined parameters such as for example the size of the particle, after interaction with the first scattered light measuring unit, decides whether the particle is or is not analyzed in the identification unit. For that purpose, reading-out is effected by way of a programmable AD-converter card with integrated processor, preferably an 80x86 processor, at a frequency of about 20 kHz, the size or the refractive index is ascertained by means of the integrated program and compared to the preset value. If the particle falls in the range which is of interest, a trigger signal is sent to the identification unit, whereupon the particle is characterized.

Use of the integrated AD-converter card gives a very high level of system safety and security. Use of the electronic circuit basically permits use of the described system in particle-rich environments in which the identification unit, without preselection, would be overloaded.

The invention will be described hereinafter by means of an embodiment. In the accompanying drawings:

Figure 1 is a diagrammatic view of the module units and the co-operation thereof, and

Figure 2 shows vibration spectra involving conventional Raman technology and NIR-Raman technology according to the invention.

The method and the device according to the invention which is used for carrying out the method are to be presented by way of example in terms of the identification of a polymer microparticle of a size of between 0.5 μm and 15 μm, which is typical for contamination in clean rooms.

Contamination in a clean room, for example of the 1,000 category, is sucked in by a pump and converted by means of a nozzle and a particle feeder 1 into an individual particle flow.

In this situation the particle 10 generally acquires a speed of approximately 10 mm/s. That particle 10 now passes the first laser beam L1 which is emitted for example by an HeNe-laser 2 with approximately

20 mW output power and focused at 50 μm. The scattered light is detected in dependence on angle and evaluated to determine the particle size in accordance with the known theory of elastic light scatter (Mie theory). If a selection of given particles is to be effected, for example in accordance with a given diameter, the laser 2, upon fulfillment of the selection properties by the detected particle, sends a trigger signal by way of a control 3 to the downstream-connected identification unit which comprises a corona discharge 4, an excitation laser 5 and a spectrometer unit 6. The selectable properties of the particles can be preselected by means of software at the electronic control 3. That selection technology is particularly advantageous in regard to use of the identification unit in particle-rich environments. If no selection is to be effected the trigger signal is sent at each detected particle 10.

After detection and determination of size by the first laser 2 the particle 10 passes the corona discharge 4 which is operated for example at 10,000 V. In this case the particle 10 is occupied with charge proportionally to the surface area. In a downstream-disposed electromagnetic field, a so-called electromagnetic brake 7, the particle 10 is decelerated to a speed of about 1 mm/s, thus affording a resonance time of the particle 10 in the second laser beam L2 of about 10 ms. The laser beam L2 is preferably produced by a semiconductor laser 5 with a wavelength of 780 nm and an output power of 300 mW and focused to 10 μm beam diameter.

The light which is inelastically scattered in that period is detected after suppression of the excitation wavelength by means of a holographic notch filter by between one and three minispectrometers 6, the geometrical arrangement of which is such that spectra are obtained with a resolution of 12 cm^{-1} over a wavelength range of 200 – 4,000 cm^{-1} relative to the excitation wavelength. The vibration spectrum obtained in that way in the range of 200 - 4,000 cm^{-1} is electronically filtered and investigated for spectral features (peaks).

The peak table obtained is finally compared to a database 8 which contains the necessary reference tables and the substance of the particle 10 identified.

Figure 2 shows the spectrum of a palmitic acid particle which was used as a test system. It is of a diameter of about 4 μm and was recorded once with a residence time in the second laser beam L2 of 10 ms and once with a shorter residence time, that is to say without braking of the particle 10. The lower spectrum shows the recording with conventional Raman technology and the upper spectrum shows that with a short exposure time of 10 ms and a resolution of 12 cm^{-1} with the procedure according to the invention on an individual particle. With a short exposure time, all spectral features can be recognized, and the noise is markedly more pronounced.

In the predominant number of cases, the signal which is obtained without use of the electromagnetic brake, that is to say without a prolongation of the available measuring time, is not sufficient for identification as the noise increases greatly and therefore automated evaluation becomes impossible.